

Table 1

<u>"Cr"(eq)</u>	<u>Ac₂O(eq)</u>	<u>H₅IO₆(eq)</u>	<u>Conc. (M)</u>	<u>yield</u>
0.10	2	3.0	1 to 0.1	87%
0.05	1	0.5	2 to 0.5	47%
0.05	TFAA (2)	0.5	2 to 0.5	34%
0.05	1	1.0	2 to 0.3	59%
0.05	1	1.5	2 to 0.2	69%
<u>0.05</u>	<u>2</u>	<u>3.0</u>	<u>2 to 0.1</u>	<u>96%</u>
0.00	2	3.0	2 to 0.2	---

Initial oxidations of ethyl benzene were carried out with excess CrO₃, but the more soluble chromoyl diacetate was found to be a superior oxidant. Other chromium[VI] substrates including chromoyl chloride, chromoyl bistrifluoroacetate, chromoyl bistriflate, and chromoyl bis t-butylester were tested but were found to be inferior to chromoyl diacetate. The premier co-oxidants tested proved to be periodic acid and tetrabutylammonium periodate. Hydrogen peroxide, t-butyl hydroperoxide, diacyl peroxides, TMSOOTMS, peroxydisulfate, and persulfate proved to be inferior co-oxidants when compared to periodic acid and tetrabutylammonium periodate.

Of considerable significance is that C-H oxidation occurred at about -40°C, and the reaction is catalytic in chromium. Reaction mixtures found to be deficient in co-oxidant at first exhibited a brownish-orange color, but then rapidly turned green and ceased oxidation. Addition of excess periodic acid re-established both the brown-orange color and the oxidative process. Both excess periodic acid and acetic anhydride were found to be required for optimal yield of acetophenone from ethyl benzene.

An expanded series of substrates listed in Table 2 below were oxidized in accordance with the same procedure. The results of such oxidation revealed several aspects of the method of the instant invention. The method is stereospecific and retains the stereochemistry of the C-H bond oxidized, as is evident from Table 2, entries 6-9 and 10. While a second oxidation can yield a *cis*-diol, periodate effects rapid oxidative cleavage, as evidenced by